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PCT Applicant's Guide - Volume II - National Chapter - US

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FORM PTO-1390 (REV 10-95)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				15675.P322
				U.S. APPLICATION NO. (If known, see 37 CFR 1.5)
INTERNATIONAL APPLICATION NO. PCT/FR99/00043	INTERNATIONAL FILING DATE January 12, 1999	PRIORITY DATE CLAIMED January 12, 1998		
TITLE OF INVENTION magnetic etching process, especially for magnetic or magnetooptic recording - UTILITY				
APPLICANT(S) FOR DO/EO/US Claude Chappert; Harry Bernas; Jacques Ferre				
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:				
<ol style="list-style-type: none"><li>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</li><li>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</li><li>3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b)) and PCT articles 22 and 39(1).</li><li>4. <input type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.</li><li>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)).<ol style="list-style-type: none"><li>a. <input checked="" type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</li><li>b. <input type="checkbox"/> has been transmitted by the International Bureau.</li><li>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li></ol></li><li>6. <input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).</li><li>7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).<ol style="list-style-type: none"><li>a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</li><li>b. <input type="checkbox"/> have been transmitted by the International Bureau.</li><li>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li><li>d. <input type="checkbox"/> have not been made and will not be made.</li></ol></li><li>8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</li><li>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).</li><li>10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</li></ol>				
Items 11. to 16. below concern document(s) or information included:				
<ol style="list-style-type: none"><li>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</li><li>12. <input checked="" type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</li><li>13. <input type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</li><li>14. <input type="checkbox"/> A subsequent specification.</li><li>15. <input type="checkbox"/> A change of power of attorney and/or address letter.</li><li>16. <input checked="" type="checkbox"/> Other items or information:  --This is a continuation-in-part of PCT/FR99/0043-- priority request; English specification (continuation in part of publication no. WO 99/35657); copy of forms PCT/IB/301 &amp; 304; request of filing; copy of int'l publication no. WO 99/35657; verified English x'lation of int'l. publ.; copy of preliminary exam report w/new page (claims 1-6); verified English x'lation of amended page 1; copy of English translation of prelim. exam report</li></ol>				

Annex US.II, page 2 PCT Applicant's Guide - Volume II - National Chapter - US

U.S. APPLICATION NO. of known, 37 CFR 1.51		INTERNATIONAL APPLICATION NO. PCT/FR99/00043		ATTORNEY'S DOCKET NUMBER 15675.P322																																																					
17. <input checked="" type="checkbox"/> The following fees are submitted: <b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5) ):</b> Neither international preliminary examination fee (37 CFR 1.482 nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by EPO or JPO ..... \$970.00 International preliminary examination fee (37CFR1.482)not paid to USPTO but International Search Report prepared by the EPO or JPO. . . . \$840.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee paid to USPTO (37 CFR 1.445(a)(2)) ..... \$760.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) .... \$670.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$96.00 <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b> \$ 840.00 Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)). \$ <table border="1"><thead><tr><th>CLAIMS</th><th>NUMBER FILED</th><th>NUMBER EXTRA</th><th>RATE</th></tr></thead><tbody><tr><td>Total claims</td><td>15 - 20 =</td><td>0</td><td>X \$18.00</td></tr><tr><td>Independent claims</td><td>1 - 3 =</td><td>0</td><td>X \$78.00</td></tr><tr><td colspan="3">MULTIPLE DEPENDENT CLAIM(S) (if applicable)</td><td>+ \$260.00</td></tr><tr><td colspan="3"><b>TOTAL OF ABOVE CALCULATIONS =</b></td><td>\$ 1100.00</td></tr><tr><td colspan="3">Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).</td><td>\$</td></tr><tr><td colspan="3"><b>SUBTOTAL =</b></td><td>\$ 1100.00</td></tr><tr><td colspan="3">Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).</td><td>\$</td></tr><tr><td colspan="3"><b>TOTAL NATIONAL FEE =</b></td><td>\$ 1100.00</td></tr><tr><td colspan="3">Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property</td><td>+</td></tr><tr><td colspan="3"><b>TOTAL FEES ENCLOSED =</b></td><td>\$ 1100.00</td></tr><tr><td colspan="3"></td><td>\$ Amount to be: refunded \$</td></tr><tr><td colspan="3"></td><td>charged \$</td></tr></tbody></table>				CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	Total claims	15 - 20 =	0	X \$18.00	Independent claims	1 - 3 =	0	X \$78.00	MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$260.00	<b>TOTAL OF ABOVE CALCULATIONS =</b>			\$ 1100.00	Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).			\$	<b>SUBTOTAL =</b>			\$ 1100.00	Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).			\$	<b>TOTAL NATIONAL FEE =</b>			\$ 1100.00	Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property			+	<b>TOTAL FEES ENCLOSED =</b>			\$ 1100.00				\$ Amount to be: refunded \$				charged \$	<b>CALCULATIONS FOR PTO USE ONLY</b>	
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a. ☒ A check in the amount of \$ 1100.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. in the amount of \$ to cover the above fees.  
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**NOTE:** Where an appropriate time limit under 37 CFR 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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REGISTRATION NUMBER

534 Rec'd PCT/PTC 12 JUL 2000

MAGNETIC ETCHING PROCESS, ESPECIALLY FOR  
MAGNETIC OR MAGNETOOPTIC RECORDING

5 The present invention relates to a magnetic etching process.

More particularly, the invention applies advantageously to ultrahigh-density magnetic recording (production of discrete magnetic materials, magnetic memory circuits, magnetically-controllable logic  
10 circuits, etc.), optical recording of the read-only memory type (CDROM, DVDROM, etc.) and production of magnetically-controllable optical circuits (diffraction gratings, photonic gap materials, etc.) using a controlled variation of the optical index component  
15 associated with the magnetism.

PRIOR ART

The extraordinary development of multimedia  
20 technologies and services in recent years has led to a race to increase the recording density. In the field of rewritable disks, although optical (phase change) technologies are developing rapidly, magnetic techniques remain the first choice, and most  
25 particularly the "hard disk", for its high transfer rate. However, the magnetic techniques ought to be limited to storage densities of 100 bits/cm<sup>2</sup>.

One of the limiting factors will especially be the transition to contact recording, for distances  
30 between the read head and the recording medium of less than 10 nm: there is a trend toward recording technologies of "the" "tunnel-effect microscopy" ("STM-like storage") or "near-field" type.

Several technological jumps have been proposed  
35 in this direction in recent years, for example near-field CD-ROM or near-field magnetooptic recording.

In this regard, reference may advantageously be made to the following various publications:

Y. Martin, S. Rishton, H.K. Wickramasinghe, Appl. Phys. Lett. **71**, 1 (1997).

5 Y. Betzig, J.K. Trautman, T.D. Harris, J.S. Weiner, R.L. Kostelak, Science **251**, 1468 (1991).

B.D. Terris, H.J. Mamin, D. Rugar, W.R. Studenmund, G.S. Kino, Appl. Phys. Lett. **65**, 388 (1994).

10 E. Betzig et al., Appl. Phys. Lett. **61**, 142 (1992).

M. Myamoto, J. Ushiyama, S. Hosaka, R. Imura, J. Magn. Soc. Jpn. **19-S1**, 141 (1994).

15 T.J. Silva, S. Schultz, D. Weller, Appl. Phys. Lett. **65**, 658 (1994).

M.W.J. Prinz, R.H.M. Groeneveld, D.L. Abraham, H. van Kempen, H.W. van Kesteren, Applied. Phys. Lett. **66**, 1141 (1995).

Reference may also be made to the publication:

20 B.D. Terris H.J. Mamin, D. Rugar, Appl. Phys. Lett. **68**, 141 (1996) in which it was announced that the company 3M would shortly be commercializing a magnetooptically-read "hard disk" using a solid immersion lens (SIL).

25 However, the main limitation of magnetic techniques should be the "paramagnetic limit", that is to say the size below which the bits will be erased by themselves due to a thermal effect.

30 In the current hard disk technology, the recording medium is a particulate material (magnetic particles in a nonmagnetic matrix, or magnetic particles (grains) separated by nonmagnetic grain boundaries (ME tape)). Now, minimization of the noise necessitates increasing the number of magnetic  
35 particles seen by the read head, while these particles must be magnetically decoupled as far as possible. The size of the particles is therefore very much less than the size of a bit. By extrapolating the current data,

the particles would become paramagnetic below 8 nm, thereby limiting the recording density to around 100 bits/ $\mu\text{m}^2$ .

In magneto-optic recording, the materials used at the present time are amorphous alloys of the rare earth/transition metal type, which could be replaced with Co/Pt multilayers or alloys with the advent of the blue laser. Bits 60 nm in size could actually be written by a thermomagnetic effect in continuous Co/Pt multilayers, but it is probable that noise problems due to the recording medium (domain stability, domain wall roughness) would intervene, at bit sizes very much greater than 60 nm.

To extend this limit, it has recently been proposed to replace the current recording medium materials with discrete materials in which the magnetic bit limits would be geometrically defined by lithographic methods:

either deposition on an etched surface,  
S. Gadetsky, J.K. Erwin, M. Mansuripur,  
J. Appl. Phys **79**, 5687 (1996)

or growth of isolated magnetic particles whose size and position are defined by lithography,  
S.Y. Chou, M.S. Wei, P.R. Krauss, P. Fischer,  
J. Appl. Phys. **76**, 6673 (1994).

The latter technique would allow there to be only a single magnetic particle per bit.

In parallel, pressing techniques based on a matrix defined by electronic lithography have been developed,

S.Y. Chou, P.R. Krauss, P.J. Renstrom, Science **272**, 85 (1996),

Y. Xia, X.M. Zhao, G.M. Whitesides, Microelectron. Eng. **32**, 255 (1996),

which, just as in X-ray or interferential lithography, could in the near future allow mass production of etched media, with patterns very much less than one micron in size over areas of a few  $\text{cm}^2$ , probably sufficient for disks of the future.

However, in the current published work, these various techniques have several drawbacks:

1. Whatever the technique adopted, recording in contact mode will require a material having a low and controlled surface roughness: the etched materials proposed up until now will therefore require a final, and probably difficult, planarization step.

2. In the case of near-field magnetooptic recording, sudden variations in optical index (variations in reflectivity) of the etched material will give diffraction effects, which may be manifested by much greater polarization variations than those induced by the magnetic domains - a source of unacceptable noise.

3. A final problem, at very high densities on these etched materials, concerns the following of the track, and it will probably be necessary to develop a specialized "track" for this purpose, but without degrading the points mentioned above.

#### PRESENTATION OF THE INVENTION

The subject of the invention is a magnetic etching process, characterized in that a thin-film magnetic material (comprising a few atomic planes) is controllably irradiated in order to locally modify, over regions having a width of the order of one micrometer or less, the magnetic properties of said material, such as, in particular, its coercivity, its magnetic anisotropy or its Curie temperature.

Such a process allows the aforementioned problems to be solved. In particular:

1. The roughness of the original film is unchanged by irradiation and can therefore be adjusted independently. In particular, it may be envisaged to carry out a postirradiation deposition (for the production of devices) under excellent growth conditions (% at an etched surface).

2. The optical index variations remain small for considerable changes in the magnetic properties and can, moreover, be controlled, within a certain range, almost independently of the magnetic variations obtained, by the structure of the substrate or the energy of the ions.

3. The effect of the irradiation is cumulative: it is possible to carry out the irradiation several times, and to obtain the same result as in a single time with the cumulative dose. This aspect may be useful when it is desired to irradiate several regions of the specimen with different values, or at different steps in the fabrication of a device.

4. The effect of the irradiation may be easily controlled in real time, by measuring the change in the properties (for example magnetic properties) over a test region.

5. The technique is easy to employ for the mass production of recording media, and to do so economically since the tools that it requires to be used are either already used in microelectronics (irradiation) or are under development (lithography by pressing in the case of large areas and of nanometric sizes, for example).

Advantageously, the irradiation is carried out by means of an ion beam.

Other technical means of energy deposition could be envisaged.

The irradiation may be carried out through a resin mask or with the aid of a focused ion beam.

The aforementioned etching process is advantageously used for the ultrahigh-density magnetic or magnetooptic recording of binary information, and especially for the production of discrete magnetic materials, of magnetic memory circuits or of magnetically-controllable logic circuits.

In particular, the aforementioned process has the advantage of making it possible to write magnetic domains of size very much less than 100 nm and whose

position and geometry are perfectly defined and therefore to maximize the signal-to-noise ratio and optimize the track-following problems, while preserving perfectly controlled surface roughness.

5           In addition, the process proposed by the invention is advantageously used for producing an optical recording of the read-only memory type (CDROM, DVDROM, etc.).

10           It is known in fact that the near-field optical recording techniques will probably have to use smooth writing materials, with a read head flying a few nm above said material (at the present time, 30 nm for a hard disk). Now, the current optical recording techniques of the read-only memory type are not  
15           satisfactory: the pressing methods, using dies, may give sizes of less than 100 nm but the recording medium which is obtained is rough; as regards the writing methods using a focused laser beam (ablation, phase change), these do not make it possible to work with bit  
20           sizes of the order of or less than 100 nm.

          Applications other than the recording of binary information may be envisaged. In particular, the magnetic etching process proposed by the invention is advantageously used for the production of magnetically-  
25           controllable optical circuits (diffraction gratings, photonic gap materials, etc.) using a controlled variation of the optical index component associated with the magnetism, for the production of sensors (hard disk read heads, etc.) or magnetic memory circuits  
30           (extraordinary Hall-effect memory, magnetoresistive memory, spin-dependent tunnel-effect memory).

          In particular, it is known that the emergence of photonic gap materials opens the way to producing optical devices and that one of the aspects to be  
35           resolved will be that of control of the device. The process proposed by the invention makes it possible, by irradiation through a mask, to manufacture a waveguide film made of nonmagnetic material, comprising a regular array of magnetic units (photonic crystal) having an



optical index which is both slightly different from that of the host material and magnetically controllable.

5 In general, the process proposed by the invention may apply whenever it is advantageous to define a magnetic element accurately, while maintaining a very high degree of planarity of the device (for example, in order to favor subsequent growth).

10 The process proposed by the invention may also be used for magnetically etching a layer already buried beneath other, insensitive layers, by adjusting the irradiation conditions. For example, and by way of nonlimiting indication, it is possible to produce electrical circuits etched in the same thin-film  
15 magnetic material, and only the important part of which will remain magnetic, the contact tracks having been made inactive by irradiation; the coercive field of a given region of a specimen may be controllably reduced so as to guarantee that the reversal of the  
20 magnetization will always occur under the same conditions, from the same site.

The process proposed by the invention may a *priori* be adapted to any material for which a minute variation in the local atomic arrangement can lead to a  
25 large modification in the magnetic properties, that is to say to transition metal alloys (e.g.: CoPt, NiFe, etc.), to rare earth/transition metal alloys (e.g.: TbFeCo, etc.) and to magnetic multilayers (e.g.: Co/Pt, Fe/Tb, etc.), without this list being exhaustive.

30 Co/Pt multilayers are materials which are potentially of interest for short-wavelength magnetooptic recording in blue light.

#### DESCRIPTION OF ONE OR MORE EMBODIMENTS

35

The process of magnetic etching by irradiation is described below in the case of magnetic multilayers irradiated by an ion beam and involves several steps, in which:

- (i) the composition and the roughness at the interfaces and on the surface of the layers are carefully controlled before irradiation;

- (ii) the multilayer structure is irradiated by an ion beam, the structural modification induced by the beam being controlled; in particular, the energy density deposited by the beam is controlled by choosing the mass and the energy of the incident ions;

- (iii) the irradiation may be completed by a suitable thermal annealing step in order to relax the stresses and/or induce local ordering.

In the case of magnetic materials, the effects of the process are important on alloys (transition metal alloys, rare earth alloys and rare earth/transition metal alloys) and on multilayer stacks of all types.

The process is advantageously employed on Co/Pt multilayers. It should be noted that these materials have already been very widely studied for their properties, firstly their perpendicular magnetic anisotropy and secondly their strong magnetooptic Kerr effect; they therefore constitute advantageous candidates for magnetooptic recording.

In materials based on ultrathin multilayer films, the properties are dominated by the competition between the interface effects and the volume properties. For example, the easy magnetization direction is given by the sign of an effective anisotropy coefficient  $K_{eff}$  which, to a first approximation, is given by:

$$K_{eff} = -K_d + K_v + \frac{(K_{s1} + K_{s2})}{t_{co}}$$

The first term represents the dipole shape anisotropy ( $K_d > 0$ ), the second term represents the volume anisotropy ( $K_v > 0$  in the case of Co) and the last term is due to the interfaces ( $K_s > 0$  in the case of the Co/Pt interface), the influence of which varies

inversely with the Co thickness  $t_{Co}$  ( $K_{s1}$  and  $K_{s2}$  denoting the magnetic anisotropy coefficients of the two interfaces of the Co film. Depending on the sign of  $K_{eff}$ , the easy magnetization axis is either the axis perpendicular to the plane of the layers ( $K_{eff} > 0$ ) or the plane of the film. The perpendicular configuration is necessary for magnetooptic recording and will probably become the standard for ultrahigh-density magnetic recording, all techniques included.

The process is preferably limited to irradiation resulting in low energy deposition (small number of atomic displacements at the interfaces that we are interested in). This may be achieved, for example, by light ions (e.g.  $He^+$ ) of low energy (from a few keV to about a hundred keV) or else by heavy ions (e.g. mass of the order of 100) of relatively high energy (typically, 1 MeV). The irradiation firstly modifies the composition of the interface and therefore, in particular, the anisotropy. For the thinnest films (1 or 2 atomic planes) or for higher doses, the composition of the film and hence its volume magnetism are also modified (by transferring atoms from one layer to another): in the particular case of Co/Pt, the Curie temperature of the CoPt alloy decreases with Pt concentration, and becomes below room temperature at around 75% Pt.

For example, the inventors have rendered specimens, having a thickness  $t_{Co}$  of 0.5 nm, paramagnetic at ordinary temperature, in a controlled manner, by irradiating, at a (very low) dose of  $10^{15}$  ions/cm<sup>2</sup>, with  $Kr^-$  ions accelerated to 300 keV, as well as with 30 keV  $He^+$  ions at a dose of  $10^{16}$  ions/cm<sup>2</sup>.

The effects of the irradiation were firstly characterized on simple Pt(3.4 nm)/Co( $t_{Co}$ )/Pt(6.5 nm)/amorphous substrate (Herasil polished silica,  $SiO_2/Si$ ,  $Si_3N_4/Si$ ) sandwiches deposited by sputtering.

With the deposition technique used, magnetic films with a perpendicular easy magnetization axis and a perfectly square polar hysteresis loop (100% remanent

magnetization) within the Co thickness range: 0.3 - 1.2 nm are obtained before irradiation.

The irradiation of these specimens at He<sup>+</sup> ion fluences up to around  $2 \times 10^{15}$  atoms/cm<sup>2</sup>, the ions being accelerated to energies of between 5 and 100 keV, makes it possible actually to adjust the magnetic properties of an ultrathin Co layer:

1. on 0.5 nm thick layers (approximately 2.25 atomic planes), the main effect is a drop in the Curie temperature, which may fall below room temperature for a dose of the order of  $2 \times 10^{16}$  ions per cm<sup>2</sup>. Below that, the film retains a perpendicular easy magnetization axis and a square loop, but the coercive field of which decreases uniformly when the irradiation dose is increased. Square magnetization loops with coercivities of a few Oe have been obtained. Advantageous applications for the production of low-field sensors may be envisaged;

2. on 1 nm thick specimens (approximately 5 atomic planes), the main effect of the irradiation is a tilt of the easy magnetization axis in the plane of the film, combined with a reduction in the interface anisotropy term  $K_s$ . The effect is obtained for low doses because the initial thickness is close to that (1.2 nm) at which the tilting effect occurs in the original specimens;

3. on specimens of intermediate thickness (0.8 nm, i.e. 4 atomic planes), the same doses have no visible effect on the hysteresis loop: at these thicknesses, the Curie temperature is already very high (close to that of bulk Co), and therefore largely insensitive to small modifications of the interface, these thicknesses also being very far from the natural thickness for tilting of the easy magnetization axis. This constitutes a useful characteristic of the process since it makes it possible, on the one hand, to irradiate a bilayer while modifying only one of the layers and, on the other hand, to work at much higher doses, more conducive to homogeneity.

It should be noted that the acceleration energy of the ions has a lesser effect on the modification of the magnetic properties than on the depthwise distribution of the level of displacements in the material. This may allow the process to be employed in thin layers buried at substantially greater depths than those used in the demonstration example.

An essential characteristic of the process proposed is that, although the effect of the irradiation on the magnetism is great, its effect on the optical reflectivity of the specimen remains small.

The contrast is invisible to the naked eye, and barely visible in a good microscope (contrast comparable to that of a domain wall in a Pt/Co/Pt specimen). The smallness of the optical effect is due to the smallness of the induced structural modifications.

Tests on (Pt/Co)<sub>6</sub>/Pt multilayer stacks were also carried out. The structures of these multilayers (thicknesses, number of Co/Pt periods) were chosen around the values normally used for magneto optic recording media. Compared with the simple picture of the variation in anisotropy with Co thickness, explained above in the case of the simple films, the effects of the irradiation on the magnetic properties are made more complex in multilayers by the magnetic interaction between the layers, which may be bipolar in origin, or an exchange interaction carried by the conduction electrons in the platinum. The latter interaction, which is actually manifested by ferromagnetism of the Pt for the interface layers, helps to raise the Curie temperature of the multilayers, especially when the Co thickness is very small. The presence of these two interactions also leads to the existence of quite a wide Co thickness range in which the system is decomposed into regular magnetic domains within which the magnetization is perpendicular ("strip" domain configuration), even for

slightly negative  $K_{\text{eff}}$  values where an easy magnetization plane would be expected.

The tests were carried out on two series of specimens, of the same Co thickness (and therefore the same single layer anisotropy) and the same number of periods, but differing in the thickness of the Pt separating layer:

A series: Pt(2 nm)/[Pt(1.4 nm)/Co(0.3 nm)]<sub>6</sub>/Pt(6.5 nm)

B series: Pt(2 nm)/[Pt(0.6 nm)/Co(0.3 nm)]<sub>6</sub>/Pt(6.5 nm)

In the case of the B series, the Pt concentration of the alloy after complete interdiffusion would be about 66% (ferromagnetic alloy) while it would be 82% for the A series (nonmagnetic alloy). On the other hand, in the B series, in which the Pt interlayer is thinner, the Co layers are more highly interacting, which in principle makes it easier to obtain the "strip" domain configuration, followed by the easy magnetization plane, by a reduction in the anisotropy.

Over the range of doses tested (up to  $10^{16}$  in the case of the A series and  $2.6 \times 10^{16}$  in the case of the B series), the irradiation results show qualitatively the same effects for both series: gradual (and easily controllable) transition from a perpendicular easy magnetization axis (with a perfectly square hysteresis loop whose coercive field decreases with the irradiation dose) to a "strip" domain configuration, and then to an easy magnetization plane. As explained above, this tilting takes place at a lower dose for the B series ( $3 \times 10^{15}$  as opposed to  $6 \times 10^{15}$  ions/cm<sup>2</sup>). At the doses used, all the specimens remained ferromagnetic at room temperature.

In all the cases described above, no variation in the surface roughness of the specimen could be detected by AFM in air, even for extremely low, of the order of 0.2 nm rms, initial roughnesses.

Tests with irradiation through a resin mask were also carried out.

On Pt(3.4 nm)/Co(0.5 nm)/Pt(6.5 nm)/Herasil simple sandwich specimens, two types of resin were tested:

1. A Shipley negative resin, suitable for submicron lithography by X-ray lithography. The resin had been deposited as a thick (0.8  $\mu\text{m}$ ) layer over only half of a specimen and then annealed under the usual conditions. The entire specimen was then irradiated and the resin removed, again under the usual conditions (hot trichloroethylene bath).

The part unprotected by the resin reproduces the effects of the irradiation that were described above, whereas the protected part shows no change in its properties. In principle, using processes already developed elsewhere, the use of the same resin, but with in addition an X-ray lithography step in order to define an array of holes therein, should at the very least make it possible to obtain arrays of magnetically etched bits 0.2  $\mu\text{m}$  in size separated by 0.2  $\mu\text{m}$ , i.e. a recording density of 25 bits per  $\mu\text{m}^2$ , almost 20 times greater than the current densities;

2. a PMMA positive resin suitable for electron lithography. The resin was deposited as a layer about 0.85  $\mu\text{m}$  in thickness and in this case was not annealed, something which might have an influence on the quality of the pattern edges. Under the standard annealing conditions for this resin (160°C, 30 min) effects start to appear in the specimens, but annealing of just as good quality is possible at lower temperatures (<120°C), at which the specimens are insensitive). Next, the specimens underwent an electron lithography step in order to define, as recesses in the resin, an array of lines 1  $\mu\text{m}$  in width, separated by 1  $\mu\text{m}$ , over an area of 800x800  $\mu\text{m}^2$ . The entire specimen was then irradiated and the resin removed under the standard conditions. Observation in a magnetooptic microscope shows that, at the chosen irradiation dose ( $10^{16}$  atoms/cm<sup>2</sup>), the irradiated part becomes paramagnetic at room temperature (this state has the

advantage of eliminating the coupling between magnetic regions). The part protected by the resin remains magnetized perpendicularly, with a square loop similar to that of the original specimen.

5 The same electron lithography process as above  
was applied to a Pt(2 nm)/[Pt(0.6 nm)/Co(0.3 nm)]<sub>6</sub>/  
Pt(6.5 nm) multilayer of the B series in order to  
create the same array of lines, followed by an  
irradiation at a dose of  $2 \times 10^{15}$  atoms/cm<sup>2</sup>. However,  
10 unlike in the case of the single 0.5 nm Co layer, the  
two parts (the protected part and the irradiated part)  
retain a perpendicular magnetization and a square loop  
with, however, a lower coercive field in the case of  
the irradiated part. In fact, observation in a  
15 magnetooptic microscope clearly shows a reversal of the  
magnetization in the reverse applied field after  
saturation, which firstly takes place in the irradiated  
lines and then propagates into the unirradiated parts  
(lines and film outside the array). In the intermediate  
20 region, magnetic domains artificially created by  
lithography are therefore obtained. Next, tests were  
carried out using near-field magnetooptic microscopy,  
which made it possible to see these artificial domains  
very precisely. This consequently demonstrates the  
25 feasibility of the proposed "contact" recording  
process. On the other hand, on specimens that were  
similar but were etched by material ablation, the same  
near-field microscopy technique reveals only the  
diffraction effects.

30 It should be noted that, after irradiation, the PMMA resin becomes more difficult to remove. Residues remaining along the features introduce roughness and a weak optical contrast of nonmagnetic origin, something which requires an additional stripping procedure in an  
35 "oxygen plasma" (a procedure well known in microtechnologies).

Finally, given the precision of PMMA-resin electron lithography, we might expect to achieve bit



sizes of less than 100 nm, i.e. a density greater than 100 bits/ $\mu\text{m}^2$ .

The techniques of the type that have just been described are advantageously used for manufacturing films which include buried magnetic structures, especially for the production of magnetically structured recording media or of magnetoelectronic devices, such as M-RAM memories, logic devices, etc.

They allow planar magnetic etching of buried magnetic layers, which does not modify the surface roughness of the material and makes it possible to control the variations in optical properties, for example to make them negligible.

These techniques can be used for mass production on an industrial scale.

Using light ions, which have no etching effect, these can be deeply implanted into the substrate, well below the layer.

The parameter is then the energy deposited per ion along the trajectory - and not the cascades of defects generated by heavy ions - thereby allowing excellent control of the electromagnetic modifications, for high doses, something which gives a homogeneous effect.

Moreover, an easy nucleation region, due to the reversal of the magnetization) and associated with phenomena occurring at the border of the irradiated region, is intrinsically obtained with the proposed technique. This is a major advantage for controlling and standardizing the magnetization reversal field in an assembly of magnetic "particles", either for a recording medium material or for a memory or logic chip, without limitation.

CLAIMS

1. Writing process, in which said material is irradiated by means of a beam of light ions, such as He<sup>+</sup> ions, having an energy of the order of or less than a hundred keV, characterized in that this material is a thin-layer material comprising buried layers deposited on a substrate, and in that one or more regions having sizes of the order of 1 micrometer or less are irradiated, the irradiation dose being controlled so as to be a few 10<sup>16</sup> ions/cm<sup>2</sup> or less, the irradiation modifying the composition of atomic planes in the material at an interface between two layers of the latter.
2. Process according to claim 1, characterized in that the irradiation is carried out through a mask.
3. Process for the magnetic or magnetooptic recording of binary information, especially for the production of discrete magnetic materials, of magnetic memory circuits or of magnetically-controllable logic circuits, characterized in that it employs a writing process according to one of the preceding claims.
4. Optical recording process of the read-only memory type, characterized in that it employs a writing process according to either of claims 1 and 2.
5. Process according to either of claims 5 and 6, characterized in that the recording material is a magnetic multilayer material, the individual layers of which are pure metals or transition metal alloys or rare earth alloys.
6. Process for producing magnetically-controllable optical circuits using a controlled variation of the optical index component associated with magnetism, characterized in that it employs a writing process according to either of claims 1 and 2.

## DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below, next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

MAGNETIC ETCHING PROCESS, ESPECIALLY FOR MAGNETIC OR MAGNETOOPTIC RECORDING

the specification of which

is attached hereto  
☒ was filed on **JANUARY 12, 1999** as PCT International  
 Application Serial No. **PCT/FR99/00043**  
 And was amended on  
 (if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above. I do not know and do not believe that the same was ever known or used in the United States of America before my invention thereof, or patented or described in any printed publication in any country before my invention thereof or more than one year prior to this application, that the same was not in public use or on sale in the United States of America more than one year prior to this application, and that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representatives or assigns more than twelve months prior to this application.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, Section 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Section 199, of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor(s) certificate having a filing date before that of the application on which priority is claimed:

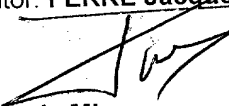
### Prior Foreign Application(s)

### Priority Claimed

<b>98/00199</b> (Number)	<b>FRANCE</b> (Country)	<b>12/JANUARY/1998</b> (Day/Month/Year Filed)	<b>XX</b> Yes	No
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

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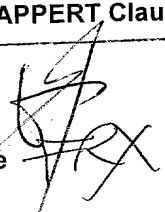
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(Application Serial No.)	(Filing Date)	(Status - patented, pending, abandoned)
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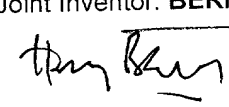
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